

DETERMINATION OF STABILISER CONTENTS IN  
ADVANCED GUN PROPELLANTS BY REVERSE PHASE  
HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

2

AD-A279 267



AR-008-634

A.R. TURNER AND A. WHITE

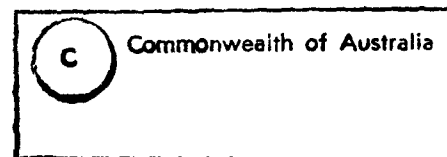
MRL-TN-663

MARCH 1994

DTIC  
ELECTE  
S MAY 17 1994 D  
F

This document has been approved  
for public release and sale; its  
distribution is unlimited.

APPROVED  
FOR PUBLIC RELEASE



MATERIALS RESEARCH LABORATORY

DSTO

THE UNITED STATES NATIONAL  
TECHNICAL INFORMATION SERVICE  
IS AUTHORIZED TO  
REPRODUCE AND SELL THIS REPORT

# ***Determination of Stabiliser Contents in Advanced Gun Propellants by Reverse Phase High Performance Liquid Chromatography***

***A.R. Turner and A. White***

MRL Technical Note  
MRL-TN-663

## ***Abstract***

*Reverse phase high performance liquid chromatographic methods for the determination of stabilisers in advanced gun propellants have been developed. The stabilisers determined were diphenylamine, 2-nitrodiphenylamine and N-methyl-4-nitroaniline. The propellants contained NC/RDX/DANPE, NC/TAGN/DANPE or NC/RDX/TAGN as energetic ingredients where NC is nitrocellulose, RDX is cyclo-1,3,5-trimethylene-2,4,6-trinitramine, TAGN is triaminoguanidine nitrate and DANPE is 1,5-diazido-3-nitrazapentane.*

DTIC QUALITY INSPECTED 3

94 5 16 058

DEPARTMENT OF DEFENCE  
DSTO MATERIALS RESEARCH LABORATORY

94-14574



*Published by*

*DSTO Materials Research Laboratory  
Cordite Avenue, Maribyrnong  
Victoria, 3032 Australia*

*Telephone: (03) 246 8111*

*Fax: (03) 246 8999*

*© Commonwealth of Australia 1994*

*AR No. 008-634*

**APPROVED FOR PUBLIC RELEASE**

## Contents

1. INTRODUCTION 5
2. EXPERIMENTAL 6
  - 2.1 *Propellant Manufacture* 6
  - 2.2 *Propellant Ageing* 6
  - 2.3 *Propellant Sample Preparation* 6
  - 2.4 *Apparatus* 7
  - 2.5 *Mobile Phase Composition* 7
  - 2.6 *Data Manipulation* 8
3. RESULTS AND DISCUSSION 8
4. CONCLUSIONS 13
5. ACKNOWLEDGEMENTS 14
6. REFERENCES 14

APPENDIX - *Reverse Phase HPLC Method for Stabiliser Determination in Advanced Gun Propellants* 15

Accession For	
NTIS CRA&I	<input checked="checked" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By	
Distribution /	
Availability Codes	
Dist	Avail and/or Special
A-1	

# ***Determination of Stabiliser Contents in Advanced Gun Propellants by Reverse Phase High Performance Liquid Chromatography***

## ***1. Introduction***

Conventional gun propellants are classified as single-, double- or triple-base. Single-base propellants contain nitrocellulose (NC) as the primary energetic ingredient, while double-base propellants contain both NC and nitroglycerine (NG) and triple-base propellants contain nitroguanidine (NQ) in addition to NC and NG.

Materials Research Laboratory (MRL) has been investigating NC-based gun propellants which contain novel energetic materials in order to obtain formulations of higher energy which offer improved ballistic performance and lower charge weights. Formulations incorporating energetic materials such as RDX (cyclo-1,3,5-trimethylene-2,4,6-trinitramine), TAGN (triaminoguanidine nitrate) and DANPE (1,5-diazido-3-nitrazapentane) have been studied to date.

Methods for the determination of stabilisers in conventional propellants by high performance liquid chromatography (HPLC) are available [1]. In the current study, HPLC methods are described for the measurement of stabiliser and stabiliser derivative levels in propellants containing the novel energetic materials described above. Reverse phase methods were preferred over normal phase methods as the solvents employed are less volatile, have lower toxicity and are lower in cost. In addition, mobile phase gradients for reverse phase HPLC tend to be less complex and have shorter run times.

Stabiliser depletion and the formation of stabiliser derivatives were measured during accelerated ageing of the propellants. Results from these studies are indicative of the stability of the propellant formulation and have been previously reported [2]. The purpose of the current paper is to describe the use of the reverse phase HPLC method to obtain stabiliser levels in these advanced gun propellants. The stabilisers involved in the current study are diphenylamine (DPA), 2-nitrodiphenylamine (2NDPA) and N-methyl-4-nitroaniline (pNMA). A formulation containing a combination of pNMA and 2NDPA was also employed to observe any synergistic effects.

---

## 2. Experimental

### 2.1 Propellant Manufacture

Batches of about 500 g of each propellant were manufactured on the MRL small scale gun propellant plant. These comprised three series of propellants: series A (NC/RDX/TAGN/Stabiliser), series B (NC/RDX/ DANPE/Stabiliser) and series C (NC/TAGN/DANPE/Stabiliser). Each series contained propellants incorporating each of the stabilisers of interest: diphenylamine (DPA), 2-nitrodiphenylamine (2NDPA), N-methyl-4-nitroaniline (pNMA) and a mixture of 2NDPA and pNMA (2NDPA/pNMA). The propellant designations and nominal compositions are given in Table 1.

The NC used was 12.6% N wood-based nitrocellulose (Australian Defence Industries). RDX was milled under water to an average particle diameter of  $\sim 5 \mu\text{m}$  and TAGN was milled under propan-2-ol to an average diameter of  $\sim 3 \mu\text{m}$ . The propellants were mixed in ethanol/ethyl acetate. A higher solvent level was required to adequately process propellants of series A which are unplasticised (250 g of each solvent instead of 180 g of each for formulations in series B and C). The propellants were extruded through a 1.14 mm die and cut to 1 mm lengths before being stored at ambient for at least two days, stoved at  $40^\circ\text{C}$  for seven days and sieved to remove undersized material.

Table 1: Propellant nominal compositions

58.5% NC (12.6% N) / 1.5% Stabiliser / 40% Other Energetic Materials:

STABILISER	SERIES A: 20% RDX + 20% TAGN	SERIES B: 20% RDX + 20% DANPE	SERIES C: 20% TAGN + 20% DANPE
1.5% DPA	A(DPA)	B(DPA)	C(DPA)
1.5% pNMA	A(pNMA)	B(pNMA)	C(pNMA)
1.5% 2NDPA	A(2NDPA)	B(2NDPA)	C(2NDPA)
0.75% 2NDPA/ 0.75% pNMA	A(2NDPA/ pNMA)	B(2NDPA/ pNMA)	C(2NDPA/ pNMA)

### 2.2 Propellant Ageing

Samples (approx 5 g) were aged for 0 - 21 days in polyethylene stoppered glass tubes (1.6 cm outside diameter x 5 cm long) immersed in a heating block regulated to  $79.9 \pm 0.1^\circ\text{C}$ .

### 2.3 Propellant Sample Preparation

Finely ground sample (0.4 g) was extracted with dichloromethane (60-70 mL) in a small-scale Soxhlet extractor for 16 hours. The extract in dichloromethane was gently evaporated on a water-bath until about 10 mL solvent remained, which was subsequently removed using a rotary evaporator. The material was transferred to a 25 mL volumetric flask with HPLC grade methanol. Internal

standard (2,4-dinitrotoluene, 3.75 mg) was added as a methanol solution and the resultant solution made up with methanol.

## 2.4 Apparatus

Reverse phase HPLC was conducted on the samples using a Waters apparatus consisting of a 600E pump, a 712 WISP autosampler, a Resolve C18 150x3.9mm column and a 490E multiwavelength UV detector operating at 270 nm. Sample injection volume was 15 µL. A detailed operating procedure is described in the Appendix.

## 2.5 Mobile Phase Composition

A ternary water/methanol/acetonitrile mobile phase was used with a flow rate of 1.2 mL/min. The mobile phase composition was a 50/10/40 water/methanol/acetonitrile isocratic mix for all samples (run time 20 minutes with 5 minute equilibration delay) except for those containing both pNMA and DANPE (ie, B(pNMA), B(2NDPA/pNMA), C(pNMA) and C(2NDPA/pNMA)). In these cases, a gradient elution program was required with a run time of 30 minutes and a 10 minute equilibration delay. This program is shown in Table 2. The composition (% water/% methanol/% acetonitrile) remains at 70/10/20 until 7 minutes. From 7 to 8 minutes, the composition is linearly ramped to 45/10/45 which remains constant from 8 to 25 minutes. From 25 to 30 minutes, the composition is linearly ramped back to 70/10/20. In a normal run (20–30 minutes run time plus 10 minutes equilibration time), the program will cycle back to the beginning after 30 or 40 minutes on another sample being injected. If the last sample has been run, the program changes the mobile phase composition to 100% methanol after 50 minutes. After 80 minutes the 600E pump shuts down and the 'program events table' linked to the gradient table turns off the helium sparge.

Table 2: Gradient mobile phase program for B(pNMA), B(2NDPA/pNMA), C(pNMA) and C(2NDPA/pNMA)

Time (min)	Flow (mL/min)	%A	%B	%C
Initial	1.2	70	10	20
7.0	1.2	70	10	20
8.0	1.2	45	10	45
25.0	1.2	45	10	45
30.0	1.2	70	10	20
35.0	1.2	70	10	20
50.0	1.2	0	100	0
80.0	0.0	0	0	0

A = water  
B = methanol  
C = acetonitrile



## 2.6 Data Manipulation

The concentration of each component in the sample solution are obtained from the chromatogram by the Waters Maxima 820 software using the following algorithm. The calibration solutions (known concentrations) are used to obtain the ratio of the response factors of component *i* and the internal standard (IS):

$$B = \frac{[i]_{\text{Calibration}} / A(i)_{\text{Calibration}}}{[IS]_{\text{Calibration}} / A(IS)_{\text{Calibration}}}$$

where square brackets indicate concentration and *A* is the response (peak area). This ratio is then used to obtain the unknown concentration of the component in the sample ( $[i]_{\text{Sample}}$ ) from the measured responses of component *i* in the sample ( $A(i)_{\text{Sample}}$ ) and the internal standard in the sample ( $A(IS)_{\text{Sample}}$ ) and the known concentration of the internal standard in the sample ( $[IS]_{\text{Sample}}$ ) according to:

$$[i]_{\text{Sample}} = B \times \frac{[IS]_{\text{Sample}}}{A(IS)_{\text{Sample}}} \times A(i)_{\text{Sample}}$$

## 3. Results and Discussion

Chromatograms from the DPA stabilised propellants of series A, B and C are shown in Figures 1, 2 and 3 respectively. In the chromatogram of A(DPA), RDX and TAGN coelute at about 2.5 minutes (Figure 1). As the concentrations of these components were not expressly required, no attempt was made to resolve the peaks due to these two components. It can be seen that the peaks for the other components are well resolved with baseline separation in all cases.

The high response of DANPE at this wavelength in the chromatograms of B(DPA) and C(DPA) (figures 2 and 3) gives large peaks which tends to overshadow the other components as all the chromatograms are scaled on the largest peak. The decrease in the RDX/TAGN peak height in the chromatogram for A(DPA) (figure 1) is attributed to the TAGN component because a decrease in the TAGN peak height is observed in the chromatogram of C(DPA) (figure 3) but no decrease is seen in the RDX peak height in the chromatogram of B(DPA) (figure 2). This is consistent with the previously drawn conclusion that there is some chemical interaction between the stabiliser and TAGN or one of its derivatives in these propellants [2].

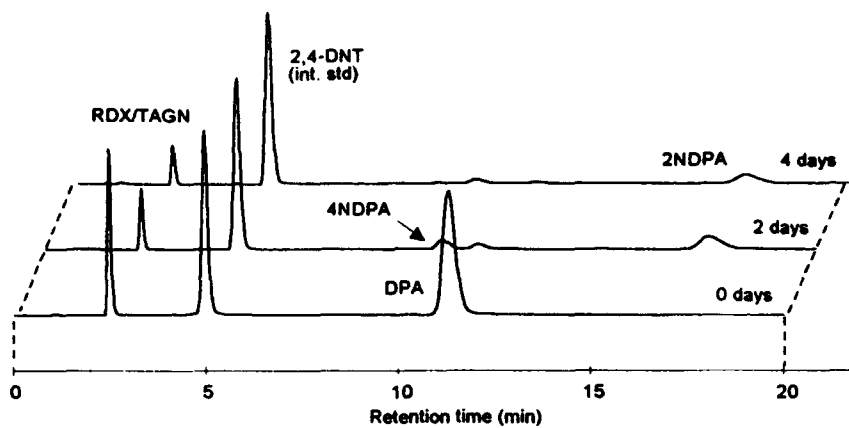


Figure 1: Reverse phase HPLC chromatograms of A(DPA) after ageing at 80°C

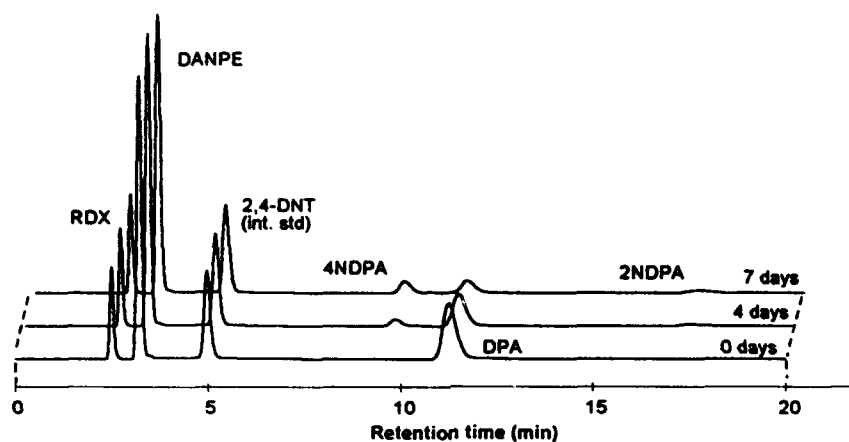


Figure 2: Reverse phase HPLC chromatograms of B(DPA) after ageing at 80°C

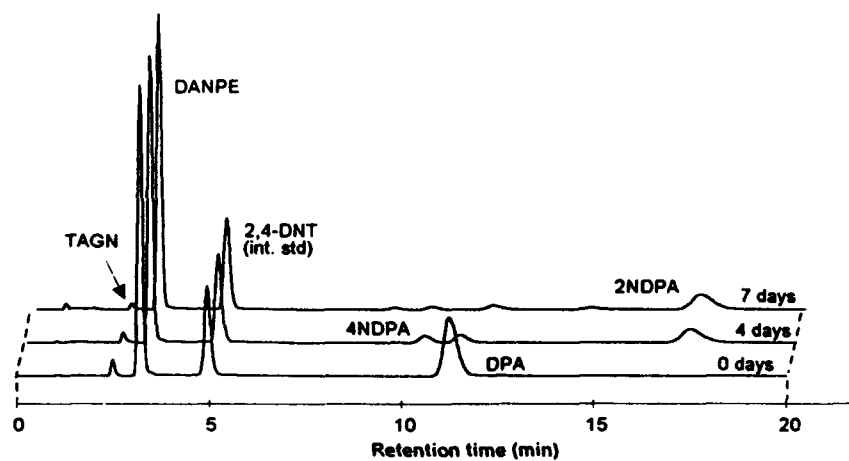


Figure 3: Reverse phase HPLC chromatograms of C(DPA) after ageing at 80°C

The chromatograms of the series A propellants stabilised with pNMA, 2NDPA and 2NDPA/pNMA are shown in figures 4, 5 and 6 respectively. The scale for the chromatogram of A(pNMA) (figure 4) is expanded as there are no peaks after about 6 minutes. Again, all the peaks are well resolved. The peaks from the two stabilisers and their derivatives in the chromatogram for the mixed stabiliser propellant A(2NDPA/pNMA) (figure 6) are well separated and so do not interfere.

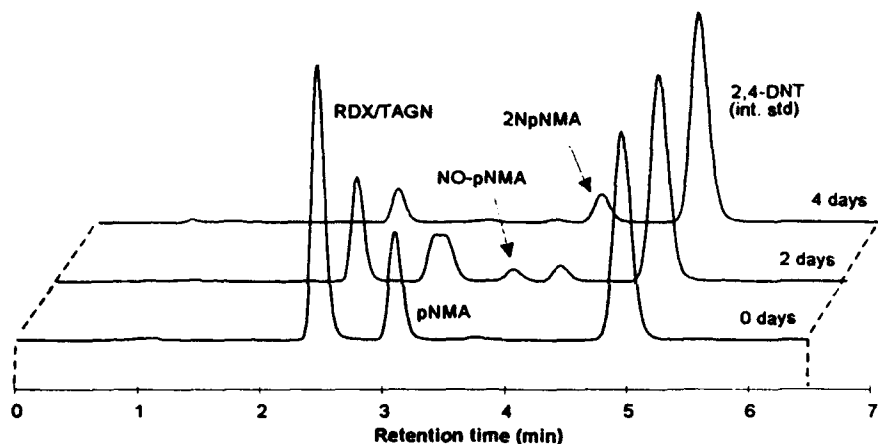


Figure 4: Reverse phase HPLC chromatograms of A(pNMA) after ageing at 80°C

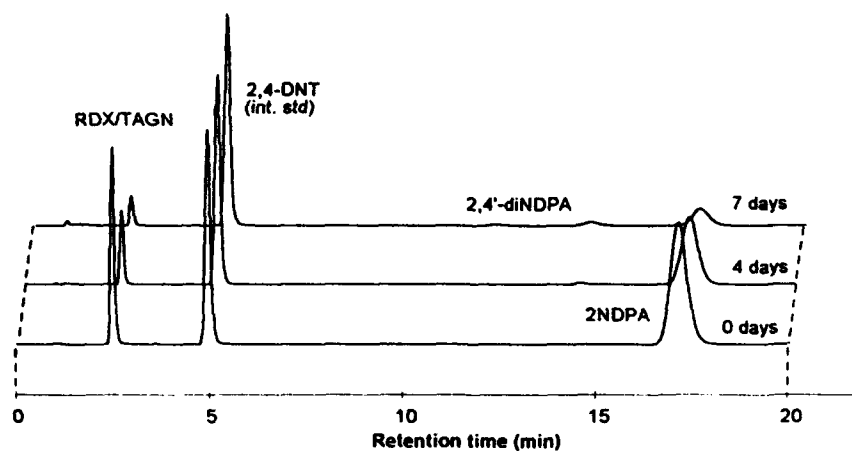


Figure 5: Reverse phase HPLC chromatograms of A(2NDPA) after ageing at 80°C

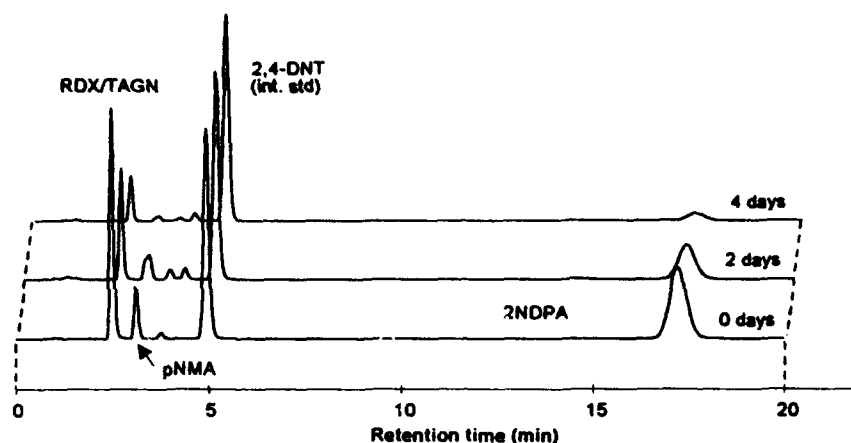


Figure 6: Reverse phase HPLC chromatograms of A(2NDPA/pNMA) after ageing at 80°C (see other chromatograms for identification of minor peaks)

The chromatograms from B(pNMA) using the gradient mobile phase program are shown in Figure 7 (only 0–20 minutes plotted). It can be seen that the elution time for a particular compound obtained using the gradient program is significantly greater than for the same compound in other samples using the isocratic program. However, the gradient program is required for samples which contain both pNMA and DANPE (ie, series B and C propellants stabilised with pNMA or 2NDPA/ pNMA) in order to separate the pNMA and DANPE peaks which would otherwise overlap using isocratic conditions.

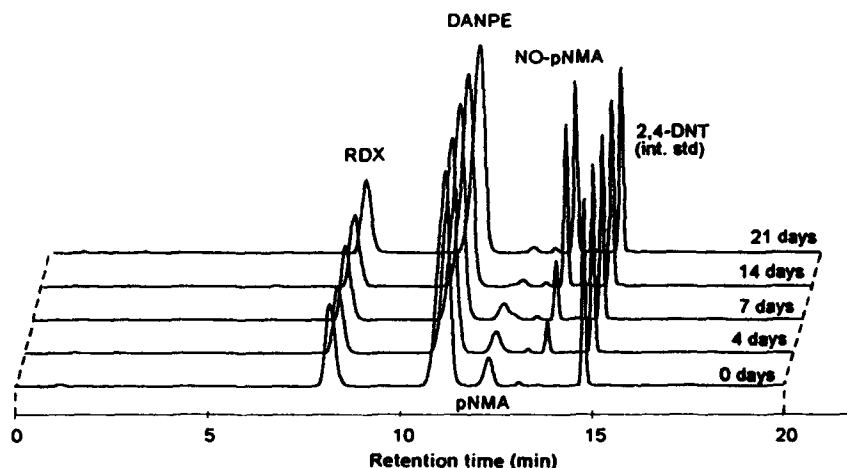


Figure 7: Reverse phase HPLC chromatograms of B(pNMA) after ageing at 80°C using gradient solvent program

As a summary, general peak retention times of the components analysed in the propellant formulations are given in Table 3.

**Table 3: Peak retention times using isocratic and gradient solvent programs**

Peak	Retention Time (min)	
	Isocratic	Gradient
RDX	2.51	8.12
TAGN	2.51	8.12
pNMA	3.06 <sup>1</sup>	12.2
DANPE	3.23 <sup>1</sup>	11.11
NO-pNMA	3.74	13.6
2NO-pNMA	4.12	14.1
2,4-DNT	4.93 - 4.94	14.8
NO-2NDPA	7.96 - 7.99	17.4
4,4'-diNDPA	9.33	
N-NO-DPA	9.61	
4NDPA	10.18	
2,4,4'-triNDPA	10.66 - 10.72	
DPA	11.14	
2,4'-diNDPA	11.81 - 11.88	19.8
2,2,4'-triNDPA	13.20	
2,2'-diNDPA	14.36 - 14.45	21.3
2NDPA	17.14 - 17.44	23.2

<sup>1</sup> pNMA and DANPE co-elute when present in the same sample.

To assess the reproducibility of the method with propellants containing new energetic ingredients, each propellant was run three times. The results are given in Table 4. It can be seen that the reproducibility is very good. Unfortunately, the actual concentrations of stabiliser in the propellants cannot be known absolutely due to losses during propellant processing.

**Table 4: Reproducibility of analyses of advanced gun propellants**

Nominal stabiliser content			Series A	Series B	Series C
1.5% DPA	Found:	Run 1:	1.37	1.35	1.37
		Run 2:	1.38	1.33	1.36
		Run 3:	1.38	1.33	1.37
		Average:	1.38 ± 0.01	1.34 ± 0.01	1.37 ± 0.01
1.5% 2NDPA	Found:	Run 1:	1.42	1.49	1.48
		Run 2:	1.42	1.48	1.47
		Run 3:	1.41	1.49	1.46
		Average:	1.42 ± 0.01	1.49 ± 0.01	1.47 ± 0.01
1.5% pNMA	Found:	Run 1:	1.42	1.45	1.48
		Run 2:	1.43	1.44	1.47
		Run 3:	1.42	1.45	1.48
		Average:	1.42 ± 0.01	1.45 ± 0.01	1.48 ± 0.01
2NDPA in 0.75% 2NDPA/ 0.75% pNMA	Found:	Run 1:	0.72	0.72	0.73
		Run 2:	0.72	0.73	0.73
		Run 3:	0.73	0.74	0.73
		Average:	0.72 ± 0.01	0.73 ± 0.01	0.73 ± 0.00
pNMA in 0.75% 2NDPA/ 0.75% pNMA	Found:	Run 1:	0.62	0.71	0.72
		Run 2:	0.65	0.71	0.70
		Run 3:	0.64	0.71	0.71
		Average:	0.64 ± 0.02	0.71 ± 0.00	0.71 ± 0.01

To assess the accuracy of the method, several solutions containing known concentrations of derivatives were made up in a similar manner to the calibration solutions but run as samples. The results are given in Table 5. It can be seen that again the reproducibility is very good, with standard deviations of less than 1%. The experimental results are within 2% of the actual values, which is considered acceptable since this figure contains all the errors from the solution preparation and analysis for both the sample and calibration solutions.

*Table 5: Results from samples with known concentrations*

Stabiliser/Method		Sample 1	Sample 2	Sample 3
DPA (Isocratic)	Found: Run 1:	4.12	6.35	9.94
	Run 2:	4.13	6.35	9.95
	Run 3:	4.11	6.36	9.95
	Run 4:	4.11	6.37	9.98
	Run 5:	4.12	6.34	9.94
	Average:	4.12 $\pm$ 0.01	6.35 $\pm$ 0.01	9.95 $\pm$ 0.02
	Known conc:	4.16	6.46	9.99
2NDPA (Isocratic)	Found: Run 1:	4.30	5.80	10.19
	Run 2:	4.30	5.79	10.20
	Run 3:	4.30	5.81	10.20
	Run 4:	4.30	5.81	10.20
	Run 5:	4.29	5.81	10.20
	Average:	4.30 $\pm$ 0.00	5.80 $\pm$ 0.01	10.20 $\pm$ 0.00
	Known conc:	4.22	5.73	10.13
2NDPA (Gradient)	Found: Run 1:	4.26	5.73	10.16
	Run 2:	4.26	5.77	10.15
	Run 3:	4.28	5.77	10.15
	Run 4:	4.28	5.77	10.09
	Run 5:	4.25	5.74	10.15
	Average:	4.27 $\pm$ 0.01	5.76 $\pm$ 0.02	10.14 $\pm$ 0.03
	Known conc:	4.22	5.73	10.13
pNMA (Isocratic)	Found: Run 1:	4.06	5.76	10.09
	Run 2:	4.05	5.74	10.05
	Run 3:	4.09	5.75	10.03
	Run 4:	4.05	5.75	10.03
	Run 5:	4.04	5.71	10.05
	Average:	4.06 $\pm$ 0.02	5.74 $\pm$ 0.02	10.05 $\pm$ 0.02
	Known conc:	4.06	5.71	10.10
pNMA (Gradient)	Found: Run 1:	4.01	5.72	10.11
	Run 2:	4.05	5.76	10.08
	Run 3:	4.08	5.76	10.10
	Run 4:	4.07	5.76	10.02
	Run 5:	4.04	5.73	10.10
	Average:	4.05 $\pm$ 0.03	5.75 $\pm$ 0.02	10.08 $\pm$ 0.04
	Known conc:	4.06	5.71	10.10

## 4. Conclusions

Reverse phase high performance liquid chromatography is a powerful technique for the rapid quantitative determination of stabiliser and stabiliser derivatives in propellants containing high energy ingredients. Isocratic conditions using a 50/10/40 water/methanol/acetonitrile mobile phase are sufficient for most propellants studied except those containing both pNMA and DANPE. In these

cases a gradient mobile phase program is required to separate the peaks of these two components.

## 5. Acknowledgments

The authors wish to acknowledge the assistance of Dr J.M. Bellerby of the Royal Military College of Science, Shrivenham, UK in the initial phase of this work. Propellants were prepared by Mr N.V. Ayres, Mr S.G. Odgers and staff.

## 6. References

- 1     *Methods of test for propellants*. DEF(AUST)5623, December 1983, Amendment No. 2, August 1993.
- 2     White, A.; Turner, A.R. and Bellerby, J.M. (1993). *Stabilisers for propellants containing new energetic materials*. Proc 24th Int. Conf. ICT, Karlsruhe, Germany.

## ***Appendix - Reverse Phase HPLC Method for Stabiliser Determination in Advanced Gun Propellants***

### ***Principle***

The prepared sample is solvent extracted with dichloromethane. The dichloromethane is removed and the extract redissolved in methanol. After addition of internal standard, the sample extract is filtered and then injected into the HPLC. The extract undergoes separation in the column of the chromatograph. The components are then eluted through the detector where they are detected. The voltage output from the detector is passed to a computer which quantifies each component using factors determined from previously run calibration solutions.

### ***Chemicals***

Dichloromethane, commercial grade or HPLC grade  
Methanol, AR grade (Ajax Chemicals -Unichrom grade specially filtered for HPLC)  
Acetonitrile 190, as above  
Distilled water, freshly distilled and filtered through a 0.45µm membrane filter  
2,4-dinitrotoluene (2,4-DNT) as internal standard  
-prepare a 250mg/100mL solution in methanol

### ***Apparatus***

Bath, boiling water  
Extractor, Soxhlet, jacketed with siphon liner and condenser to suit, Quickfit brand  
Thimble, extraction Whatman brand 18 \* 55mm or similar  
Boiling granules  
Flask, round-bottomed 100mL capacity  
Flask, volumetric 25mL capacity  
HPLC, Waters with 600E pump, 712 WISP autosampler, 490E detector, Maxima integration software  
Column, Waters Resolve C<sub>18</sub> 150 \* 3.9mm 5µm (Waters part no.85711)  
Measuring cylinder, 100mL  
Disposable filter units, 0.45µm, 25mm diameter, nylon membrane, (Activon cat.no.AGS1125N4P), 10mL glass syringes

### ***Instrument setup***

600E Pump  
Refer to Waters Operator's Manual for pump priming and purging of WISP autosampler

WISP 712 Autosampler  
After purging the autosampler place the sample carriage into the autosampler and enter the injection parameters. When all the HPLC modules are ready press the Run/Stop key to start the autosampler. The injection marker signal starts the pump's gradient timer and also starts the computer acquiring data.



#### 490E Detector

Set the required wavelength and AUFS for channel 1. Channel 3 is connected to the Omniscrite chart recorder and can be used to give a "real-time" output (use the timer to switch off the chart-recorder during an overnight run). The detector can be programmed to turn off the lamp (ie. go into standby mode) at the end of an overnight run (refer pp 4.16,4.30-31 Operator's Manual). Press the Run/Stop button to start the timer.

#### Computer/Maxima software

Refer to the Waters Maxima 820 Operator's Manual.

Load the required method.

Set up the sample queue with sample names, file names, and sample weights etc. Press F3 to start execution of the method, select [START] to ready system for background acquisition. At the end of data acquisition select [RESUME] to analyse the sample queue.

### *Procedure*

1. The sample is finely ground using an intermediate Wiley mill (glass-fronted) with a 20 mesh sieve/funnel fitted. Large-grained propellants need to be ground initially using the Wiley model 4 mill. Collect the material that passes through the 1mm sieve then regrind it using the intermediate Wiley mill. Collect all the ground material in a small stoppered vial.
2. Without delay weigh out between 0.5 and 0.6g of the prepared sample and transfer to a clean thimble (number the thimble using pencil). Record the weight of sample to the nearest 0.1mg. Place a small folded Whatman No.1 filter paper in the top of the thimble to prevent loss of sample by splashing and then place the thimble into the siphon liner of the extractor.
3. Pour approximately 60-70mL of dichloromethane into a measuring cylinder. Transfer to the 100mL round-bottomed flask. Connect the Soxhlet extractor to the flask. Connect the flask and extractor to the condenser and place on the water bath.
4. Extract the sample ensuring that the rate of reflux is such that the apparatus siphons at least every 5 minutes. Continue the extraction for a time that has been determined as sufficient for complete extraction of soluble matter.
5. Disconnect the apparatus and remove the extraction thimble allowing any remaining solvent to drain into the siphon liner of the extractor. Then carefully pour the solvent in the extractor into the round-bottomed flask.
6. Gently evaporate most of the solvent from the flask on the water bath till approximately 10mL remains. Remove the remaining dichloromethane by evaporation using the rotary-evaporator (set at 30-40°C).
7. Add 5mL of HPLC grade methanol to the flask and swirl gently to redissolve the residue. Transfer to a 25mL volumetric flask via a funnel, rinse the round-bottomed flask with methanol and add the rinsings to the volumetric flask (repeat twice). Add 1.5mL of the internal standard solution and make to volume with methanol. Filter the prepared sample solution into a Waters WISP vial using 0.45µm membrane filter.

8. Prepare a set of calibration solutions with increasing concentrations from high purity materials (for example, with concentrations similar to the typical concentrations listed below). Weigh out the required amount of the component(s) into a weighing bottle and transfer to a 25mL volumetric flask using a small quantity of methanol. Add 1.5mL of the internal standard solution and make to volume with methanol. Filter the prepared calibration solution into a Waters WISP vial using 0.45µm membrane filter.
9. Load the WISP vials into the autosampler sample carriage (calibration solutions first then the samples) then place the carriage into the autosampler.

### ***Typical calibration solution concentrations (mg/25mL)***

#### ***DPA stabilised propellants***

Component	solution			
	A	B	C	D
2,4-DNT	3.75	3.75	3.75	3.75
4,4'-diNDPA	-	-	-	2.87
N-NO-DPA	1.27	3.54	7.30	-
4nDPA	1.12	4.45	-	-
2,4,4'triNDPA	-	-	-	1.56
DPA	1.39	5.28	10.05	-
2,4'diNDPA	-	-	-	3.22
2,2'diNDPA	-	-	-	3.34
2NDPA	1.68	4.60	-	-

#### ***2NDPA stabilised propellants***

Component	solution		
	A	B	C
2,4-DNT	3.75	3.75	3.75
NO-2NDPA	2.09	4.41	-
2,4,4'triNDPA	-	-	1.50
2,4'diNDPA	6.60	1.82	3.68
2,2'diNDPA	4.92	1.64	3.16
2NDPA	2.11	5.76	10.20

#### ***pNMA and pNMA+2NDPA stabilised propellants***

Component	solution			
	A	B	C	D
2,4-DNT	3.75	3.75	3.75	3.75
pNMA	1.98	5.02	10.60	-
NO-pNMA	-	2.36	3.77	8.13
2NO-pNMA	2.52	-	-	6.45
NO-2NDPA	1.71	4.92	-	-
2,4,4'triNDPA	-	-	-	1.50
2,4'diNDPA	1.36	2.56	6.21	-
2,2'diNDPA	1.64	4.67	6.57	-
2NDPA	1.49	4.30	6.34	9.16

REPORT NO.  
MRL-TN-663AR NO.  
AR-008-634REPORT SECURITY CLASSIFICATION  
Unclassified

## TITLE

Determination of stabiliser contents in advanced gun propellants by reverse phase high performance liquid chromatography

AUTHOR(S)  
A.R. Turner and A. WhiteCORPORATE AUTHOR  
DSTO Materials Research Laboratory  
PO Box 50  
Ascot Vale Victoria 3032REPORT DATE  
March, 1994TASK NO.  
DST 91/377SPONSOR  
DSTOFILE NO.  
510/207/0027REFERENCES  
2PAGES  
18

CLASSIFICATION/LIMITATION REVIEW DATE

CLASSIFICATION/RELEASE AUTHORITY  
Chief, Explosives Ordnance Division

## SECONDARY DISTRIBUTION

Approved for public release

## ANNOUNCEMENT

Announcement of this report is unlimited

## KEYWORDS

Propellant manufacture  
Nitrocellulose  
DANPEPropellant ageing  
RDX  
RPHPLCStabiliser levels  
TAGN

## ABSTRACT

Reverse phase high performance liquid chromatographic methods for the determination of stabilisers in advanced gun propellants have been developed. The stabilisers determined were diphenylamine, 2-nitrodiphenylamine and N-methyl-4-nitroaniline. The propellants contained NC/RDX/DANPE, NC/TAGN/DANPE or NC/RDX/TAGN as energetic ingredients where NC is nitrocellulose, RDX is cyclo-1,3,5-trimethylene-2,4,6-trinitramine, TAGN is triaminoguanidine nitrate and DANPE is 1,5-diazido-3-nitrazapentane.

Determination of Stabiliser Contents in Advanced Gun Propellants  
by Reverse Phase High Performance Liquid Chromatography

A.R. Turner and A. White

(MRL-TN-663)

DISTRIBUTION LIST

Director, MRL - title page only  
Chief, Explosives Ordnance Division  
Dr R.J. Spear  
A.R. Turner 5 copies  
A. White 5 copies  
MRL Information Services

Chief Defence Scientist (for CDS, FASSP, ASSCM) 1 copy only  
Director (for Library), Aeronautical Research Laboratory  
Head, Information Centre, Defence Intelligence Organisation  
OIC Technical Reports Centre, Defence Central Library  
Officer in Charge, Document Exchange Centre 8 copies  
Air Force Scientific Adviser, Russell Offices  
Scientific Adviser - Policy and Command  
Senior Librarian, Main Library DSTOS  
Librarian, DSD, Kingston ACT  
Serials Section (M List), Deakin University Library, Deakin University, Geelong 3217  
NAPOC QWG Engineer NBCD c/- DENGERS-A, HQ Engineer Centre, Liverpool  
Military Area, NSW 2174  
ABCA, Russell Offices, Canberra ACT 2600 14 copies  
Librarian, Australian Defence Force Academy  
Head of Staff, British Defence Research and Supply Staff (Australia)  
NASA Senior Scientific Representative in Australia  
INSPEC: Acquisitions Section Institution of Electrical Engineers  
Head Librarian, Australian Nuclear Science and Technology Organisation  
Senior Librarian, Hargrave Library, Monash University  
Library - Exchange Desk, National Institute of Standards and Technology, US  
Acquisition Unit (DSC-EO/GO), British Library, Boston Spa, Wetherby, Yorkshire LS23 7BQ, England  
Library, Chemical Abstracts Reference Service  
Engineering Societies Library, US  
Documents Librarian, The Center for Research Libraries, US  
Army Scientific Adviser, Russell Offices - data sheet only  
Navy Scientific Adviser - data sheet only  
Director General Force Development (Land) - data sheet only  
DASD, APW2-1-OA2, Anzac Park West, Canberra ACT - data sheet only  
SO (Science), HQ 1 Division, Milpo, Enoggera, Qld 4057 - data sheet only  
Librarian - MRL Sydney - data sheet only  
Counsellor, Defence Science, Embassy of Australia - data sheet only  
Counsellor, Defence Science, Australian High Commission - data sheet only  
Scientific Adviser to DSTC Malaysia, c/- Defence Adviser - data sheet only  
Scientific Adviser to MRDC Thailand, c/- Defence Attache - data sheet only

Mr N.V. Ayres, EOD-S  
Mr M. Bone, EOD-S  
Mr S.P. Cashion, EOD-S  
Dr N.J. Curtis, SRL  
Mr B.L. Hamshire, EOD-S  
Mr J.F. Hooper, EOD-S  
Dr W.H. Jolley, EOD-S  
Mr R.M. Kempson, EOD-S  
Dr T. Nguyen, EOD-S  
Mr S.G. Odgers, EOD-S  
Mr L.M. Sheppard, EOD-S  
Dr S. Stephenson, EOD-S